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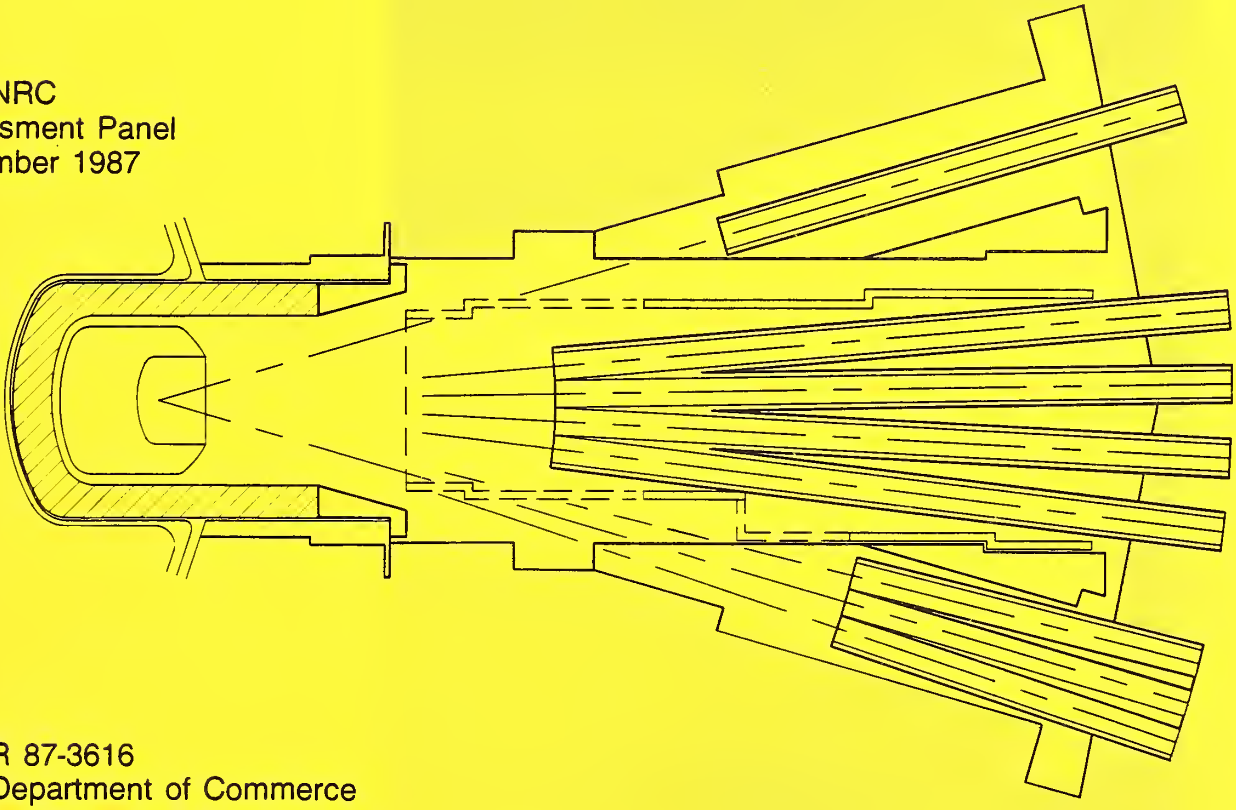
REACTOR RADIATION

NAS-NRC
Assessment Panel
November 1987



NBSIR 87-3616
U.S. Department of Commerce
National Bureau of Standards

Technical Activities 1987

Institute for Materials Science and Engineering

REACTOR RADIATION

Dr. Robert S. Carter, Chief
Mr. Tawfik M. Raby, Deputy Chief

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ABSTRACT

This report summarizes all those programs which depend on the NBS reactor. It covers the period from October 1, 1986 through September 30, 1987. The programs include the application of neutron methods to the characterization of materials, neutron standards, trace analysis by neutron activation analysis, neutron depth profiling, nondestructive evaluation, and the production of radioisotopes.

Key words: Activation analysis; crystal structure; diffraction; isotopes; molecular dynamics; neutron; neutron radiography; nondestructive evaluation; nuclear reactor; radiation.

DISCLAIMER

Certain trade names and company products are identified in order to adequately specify the experimental procedure. In no case does such identification imply recommendation or endorsement by the National Bureau of Standards, nor does it imply that the products are necessarily the best available for the purpose.

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INTRODUCTION AND OVERVIEW

REACTOR RADIATION DIVISION (460)

Robert S. Carter, Chief
Tawfik M. Raby, Deputy Chief
S. E. Tassey, Secretary

The National Bureau of Standards reactor (NBSR) is a national center for the application of reactor radiation to a variety of problems of national concern. The major areas of activity are:

Materials Characterization
Nondestructive Evaluation
Trace Analysis
Radiation Standards and Measurement

The Reactor Radiation Division (RRD), in collaboration with other scientists within the Institute for Materials Science and Engineering (IMSE), other NBS Centers, and outside organizations uses neutron scattering methods to determine the properties and behavior of materials at the submicroscopic level. These methods are used to study a wide variety of problems in such areas as hydrogen in metals, the microstructure of ceramics and metals, microscopic properties of advanced crystalline and amorphous magnetic materials and polymers, molecular species, interactions and pore structures in catalysts and microporous materials, and the structure of biological molecules. These measurements contribute to an understanding of hydrogen effects on metal properties, properties of new and improved alloys and magnetic materials, ceramics and fast-ion conductors, high-temperature superconductors, polymer blends, chemical catalysis, and biological processes. NBS is in an excellent position to carry out such a multidisciplinary program because of its strong materials programs located in IMSE and other centers and its 25 state-of-the-art reactor facilities that are not available in private-sector laboratories.

Major Activities:

The major research activities of the Reactor Radiation Division involve developing state-of-the-art neutron diffraction, inelastic scattering and radiographic methods and associated experimental facilities, and fostering their application by other NBS divisions and offices and other U.S. industrial, university and government groups to meet critical research needs in physics, chemistry, materials science, and biology. The scientific core group maintains essential research capabilities in condensed-matter science and engages in cooperative research with more than 200 scientists from NBS and outside groups, including studies of new high technology magnetic and amorphous materials, modern electronic and structural ceramics, high-temperature superconductors, chemical catalysts and advanced metals and alloys being developed for new products and technological applications.

In the area of nondestructive evaluation (NDE), RRD uses neutron radiography and both large and small-angle neutron diffraction to examine objects for defects or hidden components that must be examined nondestructively. The major effort is in the development of new or improved neutron NDE methods. Methods are being developed for such diverse applications as the use of

autoradiography to study rare paintings and the use of neutron scattering to investigate voids and other defects causing failure in structural materials.

Neutron activation analysis is a very sensitive technique for measuring trace elements at very low concentrations. RRD provides the sample irradiations, but the program and sample analysis is carried out by the Center for Analytical Chemistry (CAC). It is used extensively for characterizing Standard Reference Materials (SRM) and a variety of other measurements such as the determination of Iodine-129 concentrations and environmental studies. A large number of outside organizations also use this technique for measuring trace elements or pollutants in foods and drugs, environmental samples, criminal artifacts, etc. This is one of the largest neutron activation analysis activities in the country with thousands of samples irradiated each year. Although the primary effort in this activity is centered around neutron activation analysis, other neutron analytical methods are also being developed. A facility has been built to analyze for trace elements by measuring the prompt gamma ray spectrum induced by neutron capture in the sample, and a facility has been developed (depth profiling) to measure concentrations of light elements (e.g., B, Li) as a function of depth with ~ 100 Å depth resolution, which is the best in the United States.

A program in radiation standards and measurements is carried out by the Center for Radiation Research (CRR). Through the use of double fission chambers and a series of accurately calibrated fission foils, they provide the basis for reactor neutron flux and power density measurements needed in the U.S. fast-flux development program. The calibration and intercomparison of the series of fission foils makes use of standard reference neutron fields established in the thermal column of the NBSR. CRR also maintains well characterized, filtered neutron beams in the reactor of 2 keV, 25 keV, and 144 keV energy for the calibration and development of personnel neutron dosimeters.

A number of other groups both within and outside NBS utilize the long-term irradiation facilities at the NBSR for activities ranging from γ-ray and x-ray physics and standards to application of isotopes in medical diagnosis.

Items of Special Interest:

The installation of a large-volume cold neutron source in the NBSR in order to increase the flux of long wavelength (cold) neutrons is a high priority of NBS and the Department of Commerce. The cold source cryostat was received in November of 1986 and has successfully undergone an extensive series of tests outside the reactor. Its installation in the reactor was completed during the summer, and it is now (as of September 1987) awaiting reactor startup to complete its own startup tests.

Funding was approved by Congress in November 1986 to allow us to go forward with our program to develop a Cold Neutron Research Facility for the United States at the NBSR. Detailed design of the large guide-hall complex has been completed and a solicitation for fixed price bids was issued in August. Bid opening to grant the construction will occur in October. At the same time, the hiring or transfer of key scientific and engineering staff to carryout the planning, design, and development of the fifteen experimental stations in the guide hall is occurring. The contract for the seven guide

tubes is also close to being let. The large volume cold source will allow the provision of over 700 cm^2 of cold neutron beams (seven guide tubes, one beam hole) for cold neutron research. Ten of the major new instruments to be built in the guide hall have been identified and active design and analysis is underway for several classes of instruments. Already under detailed design and construction is the high-resolution 30 m SANS facility being jointly developed with the EXXON Research and Engineering Company.

There have been a number of accomplishments in our neutron scattering research programs during the past year, including some of the first and most accurate results (with AT&T and NRL) on the structure, oxygen arrangements, and phonon density of states in new high T_c superconductors; discovery (with U. of IL) of the existence of two distinct, magnetic propagation vectors in new, artificially produced thin-layer magnetic superlattices, along with propagation of magnetic phase information across magnetically "dead" layers; and the first direct determination (with Center for Analytical Chemistry) of the thickness and uniformity of adsorbate layers chemically bonded to the surface of microporous silica particles. In our research on hydrogen in metals and catalysts, Division scientists have revealed that the highly unusual pairing of hydrogen around metal atoms in the (hcp) rare-earths is associated with dynamic coupling modulated by one-dimensional concentration and temperature-dependent occupation correlations and have provided evidence (with the Center for Chemical Physics) for the coexistence and nature of surface and near-surface adsorbed H in a Pd black catalyst. In our theoretical efforts, we have achieved a significant advance in the theory of small angle scattering by extending the simulation technique proposed by Cahn to the treatment of scattering from bicontinuous interfaces in microdispersed and microporous media and have developed a new mathematical approach for extending the phases of diffraction data from biomolecules. In another area, the Neutron Group's Crystal Data Center has recently released for worldwide distribution (with Scientific Numeric Database of Canada) a greatly expanded crystal database with a state-of-the-art search system. Over 110 institutions, including many major industrial companies have signed up to use the system. Finally, in our new instrumentation development efforts, we have done the first experiments on the new NBS/U. of MD polarized beam spectrometer, and have made extensive use of the recently commissioned subthermal time-of-flight neutron spectrometer in studies of the dynamics of catalysts, hydrogen in metals, protonic conductors, and magnetic materials. In addition, some major components have been assembled for a modernization of the Biological Crystallography station of the NBS Biotechnology program (Center of Chemical Physics).

Reactor Utilization:

As is indicated above, RRD has a dual function. It conducts research programs in the areas of materials characterization and NDE and serves as a focal point of neutron scattering expertise for many other programs both within and outside of NBS. The second function includes not only the operation of the reactor, but also providing sample irradiation services for a large number of users.

An important part of the overall Reactor Division contribution to the NBS mission and to the scientific and technical community is in fostering the utilization of the reactor by other NBS groups and outside organizations. Interactions with other scientists and organizations take the form of both

collaborative efforts and independent programs which rely on utilization of the reactor and facilities provided by the NBSR staff. The extent of such interactions for FY 87 are indicated in the tables below. The number of personnel shown in Tables 1 and 2 include many short-term collaborators as well as permanent other agency and university guest workers. These numbers are constantly changing and so may not be exact.

Collaborative interactions are those in which workers from outside the RRD collaborate scientifically with RRD scientists on problems of mutual interest. These interactions are summarized in Table 1.

Many of the other agency, university, and industrial collaborators have worked with us regularly for many years. Others come from all over the world to spend a few days, weeks, or months to carry out specific experiments using the facilities available at the NBSR. Collaborative programs include measurements on new magnetic materials and superconductors, fast-ion conductors, polymers, catalytic materials, hydrogen embrittlement, voids and precipitates in alloys, and ceramics, etc.

Table 1. Collaborative Interactions

	No. of Personnel FY 87
RRD Permanent Scientists	17
Non-RRD Participants	
Other NBS	31
Other Agency	27
University	66
Industrial	36
International	32
Total Non-RRD	192

Independent programs are those programs carried out independently of the Reactor Radiation Division scientific staff by other NBS Divisions and outside organizations. Table 2 summarizes these interactions.

Table 2. Independent Programs

	No. of Personnel FY 87
Other NBS	30
Other Agencies	30
Universities	40
Industrial	23
International	7
Total	130

These tables demonstrate the extensive utilization of the NBS reactor by scientists and engineers from outside the Division. They come from 17 NBS

Divisions and offices, 16 other Federal organizations, 37 U.S. universities, 23 U.S. industrial laboratories, and 18 foreign laboratories.

Organization of Technical Activities:

The technical activities of the Division are organized into major tasks. These are summarized below and described in more detail in the "Description of Technical Activities" section. Because the reactor serves not only the Reactor Radiation Division and IMSE, but many other NBS Centers and outside organizations, a description of the major outside activities (Independent Programs) will also be included.

REACTOR OPERATIONS AND SERVICES

This task operates and maintains the NBSR, handles all licensing interactions, reactor security, and provides sample irradiation services to a large variety of users. Services range from pneumatic tube irradiations for neutron activation analysis to the production of radioisotopes for medical research.

NEUTRON SCATTERING CHARACTERIZATION OF MATERIALS FOR ADVANCED TECHNOLOGIES

This task develops and applies neutron scattering methods and related theoretical analysis for research on the fundamental properties of materials which affect their use in technological applications. Current areas of emphasis include new magnetic and superconducting materials, hydrogen in metals, catalytic materials, and graphite intercalation compounds. Task scientists also utilized the special role of neutrons in the structural analysis and nondestructive testing of materials, including concentrated efforts in neutron diffraction, small angle scattering, profile refinement, and state-of-the-art computer methods for: (1) precise structure and microstructure analysis for effective use of materials (e.g., in electronics, advanced transportation systems, and chemical catalysis), (2) development and transfer of an evaluated materials structure database, (3) nondestructive reference methods for texture, and strains affecting materials product processing and performance. The members of this task (and the following task) are also responsible for establishing and maintaining a national center of excellence for neutron scattering, including a computer-controlled network of nine spectrometers at the reactor.

COLD NEUTRON PROJECT

The goal of this program is to develop, install and operate a national facility for cold neutron research at the NBS reactor. This includes the operation of a large volume cold neutron source, as well as the design and construction of an extensive neutron guide tube network and a neutron guide hall with fifteen major experimental stations for research in the condensed matter and material science, biology, and fundamental physics and metrology.

INDEPENDENT PROGRAMS

Although a great deal of research by non-Division scientists is carried out in collaboration with Division scientists, there are many research projects that utilize the reactor and its services, but which are designed and carried out without any scientific collaboration with the Reactor Radiation

Division. These Independent programs typically include, trace analysis, depth profiling, radiation standards, neutron dosimetry, environmental studies, and medical research.

ORGANIZATION CHARTS AND RESEARCH STAFF

U.S. DEPARTMENT OF COMMERCE
National Bureau of Standards

DIRECTOR
Deputy Director

- Office of Congressional and Legislative Affairs
- Associate Director for International and Academic Affairs
- Associate Director for Industry and Standards

ASSOCIATE DIRECTOR FOR PROGRAMS, BUDGET, & FINANCE

- Program Office
- Budget Office
- Office of the Comptroller

OFFICE OF THE DIRECTOR NBS/BOULDER LABORATORIES

DIRECTOR OF ADMINISTRATION

- EEO Support
- Information Resources & Services
- Public Information
- Management Systems
- Plant
- Facilities Services
- Occupational Health & Safety
- Personnel
- Acquisition and Assistance

NATIONAL MEASUREMENT LABORATORY

- Office of Standard Reference Data
- Office of Measurement Services
- Center for Basic Standards
- Center for Radiation Research
- Center for Chemical Physics
- Center for Analytical Chemistry

NATIONAL ENGINEERING LABORATORY

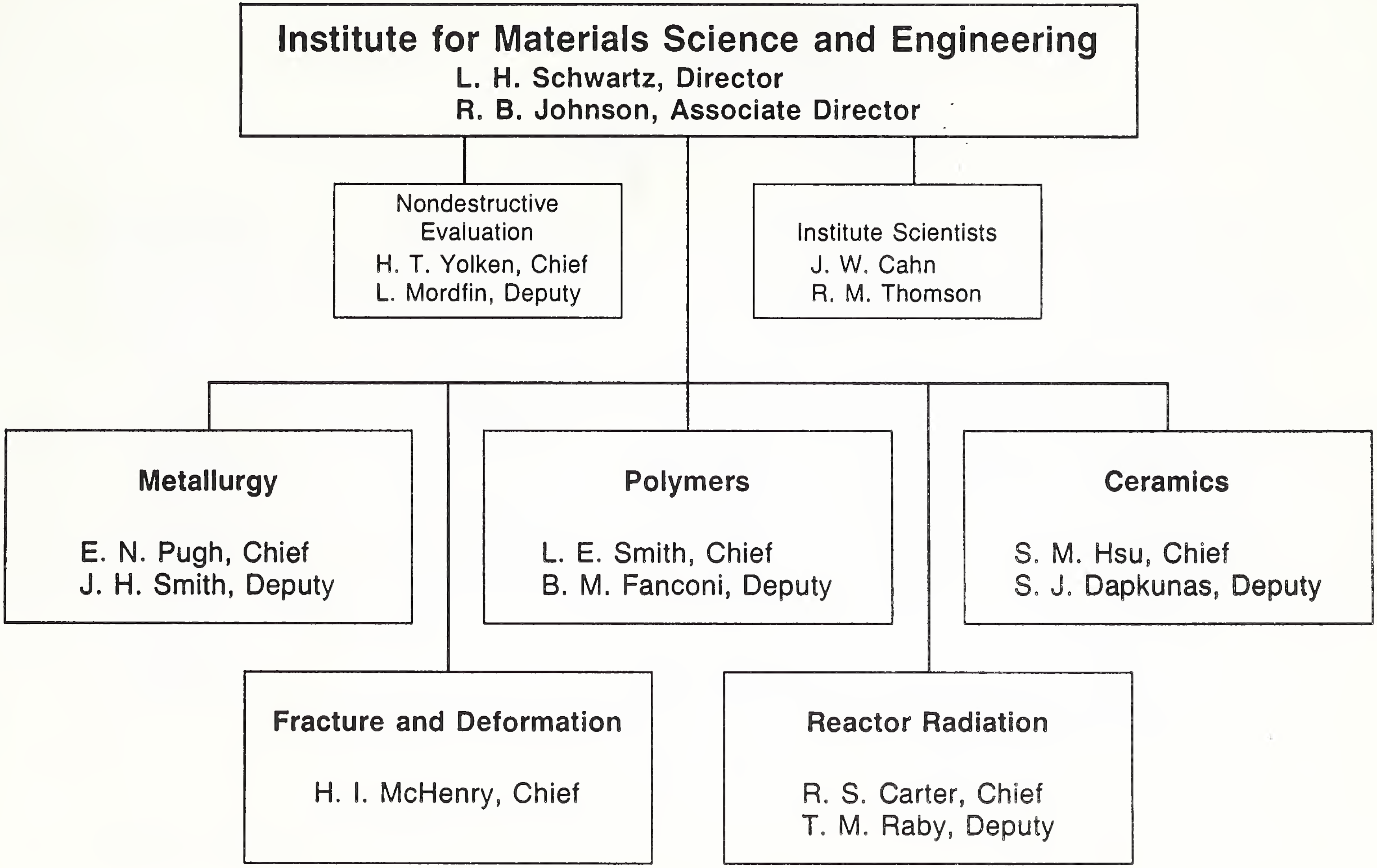
- Center for Applied Mathematics
- Center for Electronics & Electrical Engineering
- Center for Manufacturing Engineering
- Center for Building Technology
- Center for Fire Research
- Center for Chemical Engineering

INSTITUTE FOR COMPUTER SCIENCES & TECHNOLOGY

- Information Systems Engineering Division
- Systems and Software Technology Division
- Computer Security Division
- System and Network Architecture Division
- Advance Systems Division

INSTITUTE FOR MATERIALS SCIENCE & ENGINEERING

- Office of Nondestructive Evaluation
- Ceramics Division
- Fracture and Deformation Division
- Polymers Division
- Metallurgy Division
- Reactor Radiation Division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REACTOR RADIATION DIVISION
460

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M. Long, Admin. Assist.
*S. E. Tassey, Secretary
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M. Ganoczy

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B. Mozer
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J. F. Torrence, Deputy Chief
C. Harrison, Secretary

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N. Bickford
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L. Lindstrom
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W. Mueller
T. Myers
J. Ring
R. Sprow
R. Stiber
A. Toth
D. Wilkison

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J. H. Nicklas, Chief
C. Harrison, Secretary

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+J. Heine
J. Sturrock

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J. Beatty
R. Conway
R. Hayes

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#C. Choi
#V. Himes
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#J. Fernandez-Baca
#T. Giebultowicz
W. Knill
#W. Li
+J. Lynn
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J. Rhyne
#M. Spano
#Y. Yu

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D. Neumann (1/2)
#S. Trevino
R. Williams (1/2)
T. Udovic (1/2)
J. Grillo (1/2)

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D. Fravel (1/2)
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J. Gotaas
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COLD NEUTRON PROJECT

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C. Majkrzak (1/2)
D. Neuman (1/2)
I. Schroder
S. Satija (1/2)
#H. Stanley
T. Thuan
T. Udovic (1/2)
R. Williams (1/2)
D. Pierce

*Part-time
+WAE, Coop, Intermittent
#Guest Scientist, research assoc. (Full Time)

Research and Engineering Staff

N. F. Berk	o Condensed matter theory o SANS theory for microstructure analysis o Computer software for graphics and data analysis
R. S. Carter	o Reactor physics and nuclear engineering o Cold Source development
R. C. Casella	o Theory of neutron scattering from light-atom defects in metals o Group theory analyses of neutron scattering from condensed matter o Elementary particle theory, especially as related to reactor generated experiments
R. W. Erwin	o Magnetic materials o Phase transformations o Spin echo techniques o Cryogenics
C. J. Glinka	o SANS microstructure of metals and porous media o Magnetic materials o Cold neutron instrument development
J. Gotaas	o Low temperature phase transformation o SANS microstructure studies o Magnetism
P. A. Kopetka	o Mechanical engineering o Cold Source design o Electro-mechanical systems
J. LaRock	o Mechanical engineering o Neutron instrumentation design
C. F. Majkrzak	o Condensed matter physics o Polarized neutron scattering o Polarizing and monochromating devices
B. Mozer	o Structure and microstructure of metallic glasses o Dynamics of liquids o NDE of alloys
D. Neumann	o Two-dimensional materials o Solid state physics o Neutron and x-ray scattering instrumentation
J. H. Nicklas	o Mechanical engineering o Reactor fuel design o Reactor engineering support

E. Prince	o Structural properties of alloys, catalysts and minerals o Advanced crystallographic refinement methods o Software for materials structure analyses
T. M. Raby	o Reactor operations o Sample irradiations o Reactor standards
J. J. Rhyne	o Properties and transformations of high technology magnetic materials o Structure of amorphous solids o Data acquisition and analysis system
J. M. Rowe	o Orientationally disordered solids o Hydrogen in metals o Cold neutron research and instrumentation
J. J. Rush	o Hydrogen in metals o Catalysts and molecular materials o Two dimensional system
A. Santoro	o Structure of electronic and structured ceramics o Theory of crystal lattices o Powder diffraction methods
I. G. Schroder	o Cold neutron instrumentation development o Nuclear and engineering physics o Optical devices for neutron transport
S. Satija	o Low-dimensional molecular systems o Fractal aspects of microporous media o Neutron reflectometry
J. F. Torrence	o Reactor supervision o Reactor maintenance
T. J. Udovic	o Neutron time-of-flight instrumentation o Properties of catalysts o Spectroscopy of surfaces

DESCRIPTION OF TECHNICAL ACTIVITIES

The technical activities of the Division are summarized in this section. A more detailed description of each project can be found in the NBS Technical Note "NBS Reactor, Summary of Activities July 1986 through June 1987."

REACTOR OPERATIONS AND SERVICES

The NBSR is a national center for the application of neutron methods and standards to problems of national importance. The reactor provides intense neutron beams and sample irradiation facilities for more than 300 participants from many NBS divisions and outside organizations.

FY 87 Representative Accomplishments

- Nine shipments of spent fuel were made for the first time in nine years.
- The reactor control rods were replaced after more than seven years of operation.

Reactor Operations

T. M. Raby and J. F. Torrence

Reactor operations continued at the higher power level of 20 MW for most of the year. The reactor was shut down in May 1987 for installation of the Cold Neutron Source. The shutdown was used for major maintenance that will considerably reduce the length and frequency of future shutdowns. Included among these are the replacement of the shim control rods and the overhaul of the fuel transfer, both of which are complex and difficult operations. Considerable problems were encountered in the older of the two main heat exchangers. Leaks in 16 tubes suddenly developed shortly after the heat exchanger was checked and found to be free of new leaks. The heat exchanger will be monitored over the next few months to assure reliable performance. Replacement heat exchangers are being ordered, however, it will be several years before they are installed.

For the first time in several years, spent fuel was shipped for reprocessing. It took all this time to obtain an appropriate spent fuel cask and have it licensed by the Nuclear Regulatory Commission. Nine shipments were made over a period of nine weeks. This was timely because the storage pool capacity reached its limit. Again, this effort will considerably reduce future shutdowns.

During this period, the reactor was on-line almost two-thirds of the time, even with the extended shutdowns. Fuel utilization continues to be outstanding, reaching an average burnup high of 66%, by far the best in the country. Two new senior operators were licensed and a third was hired. This brings the operating staff to an acceptable level.

Irradiation Services

N. A. Bickford and J. H. Ring

Extensive irradiation services were provided to many users from within and outside NBS. In all, more than 1,500 irradiations involving thousands of samples were performed in many areas of research and application. Included among these are developments of standard reference materials, preservation and analysis of living tissue, and analysis of food and drugs and physical evidence in criminal investigations among others.

Engineering Services

J. H. Nicklas and R. S. Conway

In addition to normal engineering and design services provided to reactor operations, experimenters and users, the engineering staff is involved in a continuing effort to upgrade the reactor instrumentation, in addition to normal surveillance calibrations.

Thirty fuel assemblies have been inspected and accepted from the DOE contractor by the NBSR staff. An additional 34 fuel assemblies have been fabricated and are awaiting acceptance. Fuel compacts now being made contain 350 gms per element, a change from 300 gm elements. The first 200 plates of the 350 gm elements were radiographed and determined that the process is under control.

The fuel element handling tool heads have been modified slightly by adding another spring pin to the mounting block with bars on the lifting shaft to increase its resistance to rotation. This resistance will assure locking of the element to the upper grid and provide operators with a more positive feel when transferring fuel. A new tool for the transfer of fuel from the reactor core to the storage pool was made and tested.

The method developed to plug heat exchanger tubes with rubber plugs has been successful in reducing radiation exposures. The previous method of pounding metal plugs into the tubes required entry into the bonnet with subsequent high radiation exposure to personnel and was limited to personnel that could enter a 12-inch manhole.

A new tool for pulling the fuel transfer arm bearings which will facilitate fuel transfer has been designed and fabricated, but has not been tested to date.

The shim arm yokes were fabricated and assembled with shim arms, bearings, and splines into a complete assembly for installation into the reactor. The lost replacement of shim arm assemblies required reuse of the old yokes in a tedius underwater assembly method.

The telescopic cylinder for transferring fuel into and out of the reactor was completely rebuilt with new bearings, seals, and end caps. It was not necessary to rebuild the erecting hydraulic cylinder nor replace the connecting stainless steel lines because they were in excellent condition.

A fuel element separator was designed for installation in the licensed fuel shipping cask.

The regulation control rod was upgraded by installing a mechanical stop, replacing the multipoint plug and bearing. Special selsyn motors for the regulation rod were ordered and a new relay in the chassis of the regulation rod position indicator was installed.

A new and more reliable (personnel safety) spring compressor for the shim arm was designed and built.

The design of the auto-radiographic facility for the Smithsonian Institution was completed.

Methods were studied for plugging leaks in the thermal shield piping system. Methods were also studied for redesigning the main heat exchangers to increase their reliability.

Negotiations are proceeding with a supplier to fabricate new shaft seals for the shim arm splined shafts. The previous vendor has sold that division and no longer makes shaft seals.

The installation of a 133 KUA uninterruptible power supply for the experimental computer and associated instrumentation was completed and placed in service.

The modernization of reactor conductivity and tritium measuring instrumentation, including the installation of a new multipoint recorder in the tritium monitoring system, was completed. Six conductivity amplifiers were replaced with new units, and eleven conductivity cells and wiring were replaced.

New transmitters and venturis at the experimental level for the cryogenic facility were prepared and installed. For the bismuth-tip shield, readout meters, lights, push-buttons, and alarms, both locally on the cryogenic refrigerator and on the console of the reactor control room, were installed.

NEUTRON SCATTERING CHARACTERIZATION OF MATERIALS FOR ADVANCED TECHNOLOGIES

This task develops and applies neutron scattering techniques for precise measurement and research on the structure and microscopic properties of industrial and high technology materials which underlie their processing and use in technological applications. It establishes and maintains a national center and state-of-the-art research facilities using neutron techniques. It also fosters the application of neutron methods to NBS programs and to serve the diverse needs of the U.S. scientific and industrial communities.

Neutron beams are a powerful probe of the critical microscopic properties of materials used in the design, development and production of industrial products, in particular new materials for advanced technologies. The neutron scattering expertise and state-of-the-art, ten-spectrometer network at the NBS reactor are a central resource for 13 NBS divisions and offices and for over 50 U.S. industries and universities (over 200 participants) which need neutron techniques to address an increasing number of problems and opportunities in materials science, physics, and chemistry. Neutron scattering provides critical information on all classes of materials, which cannot be obtained by other techniques. Neutron diffraction and radiographic methods and a materials structure database are developed and maintained to meet NBS and U.S. needs for more precise structural analysis of materials. Improved capabilities will also be developed to nondestructively measure stresses and defects which affect the performance and failure of modern structural materials. Inelastic, quasielastic, and magnetic scattering methods are tailored as key probes of the submicroscopic properties of high-technology magnetic materials, industrial catalysts, and superconductors. To serve the crucial U.S. need for cold neutron measurement technology, a large cold-neutron source is being developed to be used for neutron spectroscopy and small angle scattering.

NBS scientists are currently engaged in cooperative research with industrial labs and universities on new or improved materials for semiconductor circuits, power transformers, automobiles and aircraft, sonar and microwave devices, microbatteries and fuel cells, high-strength polymers and ceramics, chemical catalysts, and microporous materials. Neutron methods are essential to many high priority NBS program areas, including surface science, biotechnology, ceramics processing, and nondestructive evaluation. Outside interactions and cooperative materials research underway includes industrial labs (e.g. Exxon, Allied, E-Kodak, IBM, AT&T, W. R. Grace, GE, GTE, DuPont, Mobil), government (NIH, Army and Navy Labs, Sandia, Smithsonian), universities (e.g. U. MD., U. Illinois, U. Calif., Carnegie Mellon, Auburn, Iowa State, Purdue, MIT). Joint research agreements or collaboration also in place with international centers: Institute Laue-Langevin, Saclay research centre, and CNRS (France), Max Planck Institute and KFA, Julich (W. Germany), U. Antwerp (Belgium), Chalmers Institute (Sweden). The materials structure database serves a nationwide user group from industry, universities and government, and has joint agreements with data centers around the world.

FY 87 Representative Accomplishments

o Scientists in the Reactor Radiation Division, in collaboration with AT&T Bell Laboratories, NRL, and the Ceramics Division, have provided some of the first and most accurate results on the atomic scale structure, oxygen

arrangements, and phonon density of states in new high T_c superconductors. Key information has also been obtained on the oxygen chains whose removal is associated with the loss of superconductivity.

- A significant advance in the theory of small-angle scattering has been achieved by the mathematical treatment of scattering from bicontinuous interfaces in microdispersed and microporous media. Based on a simulation technique proposed by J. W. Cahn in the context of spinodal decomposition, these methods provide the first topologically well-defined description of scattering from diverse physical systems united by having random morphologies with a definite length scale. The theory should provide new insights into the microstructure of technologically important materials as diverse as oil and water microemulsions and controlled-pore glasses.

- Scientists from the Reactor Radiation Division and the University of Illinois have discovered the existence of two distinct magnetic propagation vectors in artificial superlattices of erbium and yttrium produced by molecular beam epitaxy. These results also reveal the propagation of magnetic phase information across the magnetically "dead" layer of Y and showed the strong influence of "lattice clamping" on the magneto-strictively driven phase transitions occurring in this new class of tailored magnetic materials.

- High-resolution neutron vibration spectra over a wide range of H concentration for H dissolved in hcp yttrium have revealed that the highly unusual pairing of hydrogen around metal atoms in the rare-earths is associated with dynamic coupling which is modulated by concentration and temperature dependent occupation correlations along the c-axis.

- A new mathematical approach, making use of the principle of maximum entropy, has been developed for extending the phases of diffraction data from biological macromolecules from a low-resolution starting set to higher resolution. This procedure has broad potential for facilitating the determination of the structures of larger macromolecules.

- The Neutron Group's Crystal Data Center has collaborated with the Scientific Numeric Database of Canada to make the recently released, greatly expanded NBS Crystallographic Database (1.7 million records) available worldwide through CRYSDAT, an on-line, state-of-the-art database search system. Currently, over 110 institutions, including many major U.S. industrial companies, have signed up to use CRYSDAT.

- Contrast variation small angle neutron scattering (SANS) measurements have provided the first direct determination of the average thickness and degree of uniformity of adsorbate layers chemically bonded to the internal pore surfaces of microporous silica particles. The SANS results are being used to relate the morphology of the bonded phases to trends observed when such bonded phase particles are used in high-performance reverse-phase liquid chromatography.

- New insight has been gained into the origin of harmonic spin waves propagating in dilute magnetic semiconductors by workers from the Reactor Radiation Division and the University of Notre Dame. These materials are protypical examples of new perfect exchange "frustrated" coupled systems.

The work has also given the first direct determination of the isolated pair exchange energy in a series of Zn-Mn Chalcogenide magnetic semiconductors.

- In an extensive neutron-scattering study of hydrogen adsorption on the surface of Pd particles, in collaboration with the Center of Chemical Physics, neutron group scientists have found direct spectroscopic evidence for the existence and nature of subsurface hydrogen which has been predicted by theory and other experiments to be present along with surface species in a number of metals.

1. <u>Microscopic Properties of Molecular Materials and Metallic Systems</u>

R. C. Casella, D. Neumann, J. J. Rush, W. Rymes, S. F. Trevino[1], J. Nicol[2], T. Udovic

[1]Guest Scientist, Army Armament Munitions and Chemical Command
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<u>Catalysts and Hydrogen in Metals</u>

In our joint research on catalysts with the Surface Science Division, we have completed an extensive study of the interaction of hydrogen and oxygen with the surface of Pd particles ("cleaned" Pd black) by neutron vibrational spectroscopy. An analysis of the energies and intensities of the observed vibrational peaks, along with isotope dilation spectra, indicate that the hydrogens are adsorbed on regular triangular sites on (111) surface planes and that very strong H-H dynamic interactions are present on the surface. In addition, a careful study of a lower energy feature in the measured spectra suggests that it is due to the presence of hydrogen bound in subsurface layers at sites which are octahedrally coordinated. The existence of such subsurface species has been predicted by theory and also invoked to explain the results of macroscopic surface experiments under high vacuum on palladium and other metals. In collaboration with scientists from DuPont and University of California (S.B.), we have also completed the analysis of an extensive study by neutron inelastic and quasielastic scattering, and neutron diffraction of the deammoniation process in the catalyst zeolite-rho. There is considerable interest in the nature of the bonding and dynamics of protons and molecular species in this zeolite and their relationship to subtle changes vs. temperature in the framework geometry. Our neutron studies have provided information on the details of reorientation of NH_4^+ ions bound in the cages, and on significant changes in the framework breathing modes and NH_4^+ rotational modes which occur as a function of temperature and ammonia concentration. Recent neutron studies involving <u>re</u>-ammoniation of the zeolite suggest a significant difference in the dependence of framework breathing modes on ammoniation when compared to the deammoniation experiments.

Other neutron scattering research underway on catalytic systems include studies with Auburn University of hydrogen chemisorption on RuS_2 catalysts used in de-sulfurization processes and low-energy spectroscopic studies of the dynamics of adsorbed molecular hydrogen in Co-Na-A zeolite.

Our research this past year on hydrogen in metals has concentrated on the study of unusual bonding states of dilute hydrogen in rare earth metals and

in β-phase vanadium hydride. The BT-4 spectrometer continues to be the most sensitive in the world for studies of vibrational dynamics of dilute metal-hydrogen systems and for isotope-dependent spectroscopy. We have extended our previous study of the novel pairing of protons around metal atoms along the c-axis in hcp yttrium to a careful measurement of vibrational energies and lineshapes as a function of hydrogen concentration. These results, along with an investigation of dynamic interactions between protons by isotope dilution spectroscopy, clearly demonstrate that these unusual pairing interactions are dynamically coupled across the metal atoms and are modulated by longer-range concentration and temperature-dependent occupations along the c-axis.

In our study of the metal-hydrogen potential of β-phase vanadium hydride (whose vibrational dynamics exhibits both the lowest, 55 meV, and highest, 220 meV, optical phonon modes ever observed in a hydride), we have demonstrated by measuring spectra on V_2H, V_2D and V_2T that recent theoretical models to account for the highly unusual anharmonic H potential in the ab plane of the pseudo tetragonal structure are inadequate. Our results show that there are highly complex interactions between the low energy optic modes and the acoustic vibrations (band modes), which make the potential both isotope and temperature dependent. Other systems which have been investigated recently include the study of H influenced by substitutional or interstitial defects in Pd $Sc_{.02}$ and Pd_4Si glass.

Two-Dimensional Systems and Disordered Alloys

During the past year, we have also continued our work on the structure and dynamics of two-dimensional potassium-ammonia solutions intercalated in graphite being done in collaboration with scientists from the University of Illinois, Michigan State University, and the Institute Laue-Langevin. These studies have revealed that above the intercalate melting transition the ammonia molecules form four-fold complexes with the K ions which reorient about the graphite c-axis and that the ammonia molecules display essentially "free" rotations about the three-fold molecular axis. Below about 185 K the intercalate undergoes a transition to a modulated, solid phase, the exact nature of which is still under investigation. Quasielastic scattering studies of this phase have shown that while reorientations of the complexes are not present in the solid intercalate phase, the rotations of ammonia molecules persists to about 50 K, where rotational tunneling develops. In addition, inelastic scattering measurements on both phases have revealed a strong interaction between a rotational modes of the ammonia molecules and the out-of-plane phonons.

In the past year, analysis was completed on measurements reported last year of the vibrational density of states of both the crystalline and the icosahedral phases of Al_4Mn in collaboration with scientists from the University of Illinois, and the results were published. The principal conclusions of this work were: at low energies the density of states of both phases display nearly identical $g(E) \propto E^2$ dependences indicating that the two phases are elastically similar contrary to some predictions; in the intermediate energy regime (20-35 meV) the density of states of the crystalline phase shows some weak structure, while that of the icosahedral phase remains smooth; and above 40 meV there is an excess g(E) for the icosahedral phase compared to the crystalline phase. These results provide a direct test for models of the interatomic forces and dynamics of the

icosahedral phase. In another study of glassy GeSe and SiTe alloys, we have, in collaboration with Ames Laboratory and Ohio University, obtained evidence for very low energy "floppy" modes predicted for "under constrained" glass systems.

It has recently become possible to synthesize pillared clays with a wide variety of gallery heights using in situ oxidation of metal cluster cations. These systems then exhibit zeolite pore sizes and adsorption properties, thus holding out the promise of fabricating pillared clays which have catalytic properties tailored to a given chemical reaction. To date, however, little is known about the nature of the pillaring species and their interaction with the silicate layers. We have thus begun collaborative efforts with scientists from Michigan State University, SUNY at Binghamton and Schlumberger-Doll research aimed at elucidating the details of the interactions between the pillars and the host and how these interactions are modified in the presence of adsorbed species.

2. Microscopic Properties of Magnetic, Superconducting, and Amorphous Materials

J. J. Rhyne, R. W. Erwin, T. Giebultowicz[1], J. Lynn[2], M. Spano[3], J. Fernandez-Baca[3], S. C. Yu[3], and W. H. Li[3]

[1]University of Notre Dame
[2]Consultant, University of Maryland
[3]Guest Scientists

This continuing effort provides a focus for the development and application of neutron elastic and inelastic scattering techniques for fundamental studies of the microscopic magnetic properties of new classes of materials (including layered magnetic systems, rare earth compounds, magnetic semiconductors, magnetic superconductors, amorphous magnetic alloys, and spin glasses), many of which exhibit novel properties of potential use in device applications and high technology products. The effort this year was expanded to include studies of the statics and dynamics of the new high T_c superconductors. This research is carried out in collaboration with many industrial labs and universities, and the research leader was an invited speaker at the Federal Conference on Commercial Applications of Superconductivity.

During the year a number of research areas have been stressed including: (1) the determination of magnetic and magnetostrictive effects influencing the ordering of metallic superlattices produced by molecular beam epitaxy, (2) the determination of the vibrational density of states in the new high T_c superconductor $YBa_2Cu_3O_7$ and the role of the "chain" oxygen in these excitations, (3) the determination of the effects of exchange and crystal field interaction effects in dilute alloys of rare earths and yttrium, (4) the demonstration of spin-wave like modes in dilute magnetic semiconductors in the absence of long range order, (5) the discovery of two-dimensional magnetic order in the high T_c superconductor $ErBa_2Cu_3O_7$. The following summarizes some key achievements of this research:

- Established the phase coherence of magnetic order in artificial metallic superlattices (multilayers) of dysprosium and yttrium via the formation of a spin density wave. The magnetic coupling is carried from the

magnetic Dy layers through the "dead" yttrium layers with a length scale found to be inversely proportional to the thickness of the intervening yttrium layers.

- Determined quantitatively the effect of the "lattice clamping" effect on the helical-ferromagnetic phase transition in dysprosium-yttrium multilayers and in thin films of dysprosium metal. This was the first controlled experimental test of a theory of the magnetostrictive origin of the spontaneous helical to ferromagnetic moment collapse observed in rare earths.

- Established the validity of the existence of "quasi-harmonic" magnons in dilute semi-magnetic semiconductor materials which exist in these materials in the absence of long range order. Inelastic scattering data were shown to be in agreement with a calculational model involving nearest neighbor and next-nearest neighbor antiferromagnetic couplings in a ratio of 1:10 demonstrating the protypical frustration in these systems.

- Developed a procedure for determining magnetic states utilizing the change in phase of a polarized neutron beam in transmission and have applied the technique to several re-entrant spin glass systems and coexistent magnetic superconductors. These measurements were carried out using a new polarized beam instrument jointly developed with NBS and NSF funding through the University of Maryland.

- Discovered the existence of a time-dependent relaxation effect in the magnetization of single crystal alloys of the rare earths dysprosium and yttrium not previously seen in analogous alloys not possessing crystal field anisotropy. The existence of long range order in a very dilute $Y_{0.97}Dy_{0.03}$ was established and the temperature of the order parameter determined with a zero temperature value nearly equal to the free ion value of Dy.

- Discovered the antiferromagnetic ordering of erbium in the high T_c superconductor $ErBa_2Cu_3O_7$ at 0.6 K. The ordering is dipolar-coupling induced and of two-dimensional character, reflecting the highly dissimilar c-axis and basal plane lattice parameter of the Pmmm orthorhombic cell.

- Determined the one-phonon vibrational density of states in the new high T_c superconductor $YBa_2Cu_3O_7$ and demonstrated that little change occurs in these energies with temperature. A complimentary measurement of the semiconducting analog compound $YBa_2Cu_3O_6$ was made to determine the energies associated with the Cu-O breathing modes of the b-axis chain sites. It is the occupation of O in these chains which is closely tied to the occurrence of super conductivity and the high value of T_c.

- Established that superconductivity, induced at elevated pressure, is suppressed by antiferromagnetic correlations in the compound $Tm_2Fe_3Si_5$. A careful search for ferromagnetic order using polarized beam techniques showed that no such orderings occur and that the antiferromagnetic order which sets in at 1.1 K is indeed responsible for the destruction of the superconductivity in contrast to current theoretical predictions.

3. Neutron Diffraction Methods and Applications

E. Prince, A. Santoro, B. Mozer, H. Prask[1], C. Choi[1], F. Beech[2], and T. Miraglia[3], D. Tranqui[3], R. C. T. Slade[4]

[1]Guest Scientist, Armament Munitions and Chemical Compound
[2]Guest Scientist, University of London
[3]Guest Scientist, CNRS, Grenoble, France
[4]Guest Scientist, U. of Exeter

Research staff and guest scientists in this group continue to carry out a wide range of crystallographic research, including method development and applications in materials science and an International Crystal Data Center. Work involves strong collaboration with all other divisions in the Institute for Materials Science and Engineering, the Center for Chemical Physics, and with many universities and industrial laboratories, in a number of diverse research areas, including advanced ceramics for electronic or structural applications, catalysts, aerospace alloys, quasicrystals, and biological structure. Development of improved methods for residual stress and texture measurements in alloys, ceramics and composites is also a key activity.

A large component of the crystallographic effort in the winter and spring of 1987 (before shutdown in May to install the cold source) was devoted to studies (in collaboration with AT&T Bell Labs, NRL, U. of Maryland, and the Ceramics Division) on the new high-temperature superconductors. These efforts provided timely and accurate information on the oxygen chains whose occupation is associated with the onset of superconductivity, on local disorder reflected by the large temperature factors derived for these oxygens, and on the tetragonal-orthorhombic phase transformations in these materials. In addition, recent studies have been directed toward an examination of the similar ceramic structures with higher oxygen content, which also exhibit high T_c superconductivity, and toward a careful analysis of subtle structural changes which occur in nonsuperconducting compounds whose crystal chemistry is of interest for the understanding of the superconducting phases.

Our efforts in studies of structural arrangements in ionic conductors, catalysts, and biological macromolecules have continued. For example, a study of the ionic conductor system $Li_{1+X}Ti_{2-X}In_XP_3O_{12}$ revealed that there are three distinct phases: for $0 \le X \le 0.4$, the structure is of the NASICON type, and it displays similar ionic conductivity; for $0.4 \le X \le 1.0$, the structure is orthorhombic; for $1.0 \le X \le 2.0$ the structure is monoclinic. The latter two phases have substantially poorer conductivity than the NASICON phase. In a study of $V_9Mo_6O_{40}$, a phase in a system that is important because of its catalytic properties, it was found that one metal atom and one oxygen atom were disordered about a special position. In a study of lithium-exchanged zeolite X catalyst, the lithium was shown to occupy several extra framework positions that had been postulated but not previously observed. As part of our biological structure research in collaboration with scientists at the Chalmers University of Technology in Sweden, a procedure that is much more efficient than any previously proposed was discovered for applying the principle of maximum entropy to the determination and extension of the phases of diffraction data in biological macromolecules.

Finally, in somewhat different crystallographic applications, we have (with the Metallurgy Division) extended our x-ray and neutron diffraction analysis of the structure of Al-Mn-Si quasicrystals using Patterson autocorrelation functions and six-dimensional Fourier analysis; and (in collaboration with the Army and the Ceramics Division) we have applied energy-dispersive neutron diffraction techniques to a measurement of internal residual stress in both components of Al_2O_3/SiC composite materials.

NBS Crystal Data Center

A. D. Mighell, J. K. Stalick, V. L. Himes[1], M. Mrose[2], and D. R. Anderson[3]

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The NBS Crystal Data Center is concerned with the collection, evaluation, and dissemination of structure data on solid-state materials. The Data Center maintains a comprehensive database with chemical, physical, and crystallographic information on all types of well-characterized substances. These materials fall into the following categories: inorganics, organics, organometallics, metals, intermetallics, and minerals. During the year, the database and specially designed scientific software have been made available to the scientific community in three distinct modes: 1) the NBS CRYSTAL DATA Distribution Package (87); 2) International online Search System; 3) Specialized Database for electron diffractionists.

The NBS CRYSTAL DATA Distribution Package includes the database NBS CRYSTAL DATA, a FORTRAN program (NBS*SEARCH), and accompanying documentation. NBS CRYSTAL DATA contains up-to-date chemical and crystallographic data on more than 120,000 materials. Each entry consists of the reduced cell and volume, crystal system, space group symbol and number, chemical name, chemical formula, literature reference, and other data. NBS*SEARCH software has been designed to be used in conjunction with the database for the characterization and identification of crystalline materials. This product is being distributed to the scientific community by the JCPD--International Centre for Diffraction Data.

The data are available internationally through the online CRYSTDAT Search System. Within CRYSTDAT, especially designed scientific, database management and computer systems software have been integrated to form a unified analysis system. During the year, the system has been extensively upgraded with respect to data and software. Currently, over 110 institutions, including many major U.S. industrial companies, have signed up to use CRYSTDAT. Over the past months, the system has been used intensively in conjunction with the development of technologically important materials. In the design of superconductors and lasers, the database has been systematically searched for candidate compounds with the appropriate lattice parameters, space group, and chemical composition. This system was created through a collaborative effort between the NBS Crystal Data Center and CISTI's CAN/SND Scientific Numberic Database Service (Canada).

A new database, designed specifically for electron diffractionists, has been prepared. This product allows the experimentalist to identify materials

using such typical electron-diffraction data as elemental information and d-spacings. The central step in preparing this new database was the calculation of d-spacings for all inorganic compounds in NBS CRYSTAL DATA followed by compacting this information into a streamlined Search File. The product is being integrated with the software associated with commercial electron microscopes. Extensive tests using observed electron diffraction data have proved that the product provides a major new analytical tool for electron diffractionists. The generation of the electron diffraction database was carried out as a collaborative effort between NBS and the Sandia National Laboratories.

Radiographic Methods and Standards

R. S. Carter, M. Ganoczy, Y. J. Cheng[1], and J. S. Olin[1]

[1]Research Associate, Smithsonian Institution

Cooperation continues with the Smithsonian Institution in the study of works of art by neutron autoradiography techniques. It is planned that the collaborative project will involve the construction of a large enclosure to make it possible to autoradiograph large paintings at the NBSR. One great strength of neutron radiography is that it stands alone as the only available method that allows the observation of multiple pigment applications beneath the surface of an oil painting. It thus offers art historians and conservators a very special opportunity to observe the working method of an artist, quite literally from the ground up. Great progress was made during the past year, e.g., in an ongoing systematic study of the works of the prominent turn-of-the century American Impressionist, Thomas Wilmer Dewing. The work so far has provided a number of insights into the techniques of this artist over his career. Each of the five paintings radiographed appear to have been initiated and constructed in a different manner, varying from a line drawing, to several stages of painting over, to a premier coup with no preparation at all. This evidence related to the evolution of technique, along with detailed information on pigments and their origin, offer an increasingly powerful probe to improve understanding of the art of master painters here and abroad, while providing a valuable evaluation tool for judging the authenticity of works of art.

A neutron radiographic study was also conducted at the NBSR thermal column on twenty-one Tibetan bronzes from the National Museum of Natural History collection. These bronzes have high-lead alloying, thus rendering them unsuitable for studying by conventional x-ray techniques. Neutron method offers a unique nondestructive means to examine these types of objects. The neutron radiographs revealed, in detail, the sealed contents in the hollow bodies of these religious figures.

4. Small Angle Neutron Scattering

J. A. Gotaas, C. J. Glinka, N. F. Berk, S. Krueger[1], J. LaRock

[1]Guest Scientist, University of MD

Until the reactor shutdown for the installation of the cold source, the SANS instrument was utilized by experimenters for more than 98% of the available beam time, including visiting scientists from universities (22), industry

(12), government laboratories (4) from seven NBS divisions (18). The areas of experiments included polymers (44%), chemistry and physics (16%), magnetism (15%), ceramics (9%), metallurgy (7%) and biology (6%). As usual, the demand for beam time continued to be greater than the amount available. A brief review of some of this work follows:

As can be seen from the distribution of beam time given above, research in polymers is one of the most active programs on the SANS, with scientists from the Polymers Division playing a key role. C. Han (Polymers Division), with a number of collaborators from university, industry and NBS, has continued detailed studies of the conformations of a variety of polymer blends and of the critical behavior of low density polymer materials, and has examined the phase diagram and interaction parameters of rubber/rubber mixtures. In collaboration with V. Soni (Shell), Han has also used SANS to characterize the formation of micelles in block copolymers for emulsion and oil recovery applications. W. Wu and B. Bauer (Polymers Division) have continued their extensive studies of epoxy networks, determining the fractal dimension of networks and the intranetwork form factor as a function of molecular weight, as well as elucidating molecular mechanisms for the growth and deformation of epoxy networks. B. Bauer and R. Briber (Polymers Division) have studied the phase diagram of crosslinked polymer blends. Finally, M. Landry (Kodak) has used SANS to characterize both bulk polystyrene and polystyrene blends in the vicinity of the glass transition temperature.

The most active research effort from industry has been that of Exxon, which has involved a number of Exxon scientists along with collaborators from universities and NBS, and has formed a substantial part of the research in chemistry and physics. S. Sinha and J. Huang (Exxon) with H. Stanley (U. Maryland) have studied the phase separation of binary fluids, including the effects of adsorption on surfaces, and have characterized the structure of heavy oils. R. Overfield (Exxon) and H. Stanley have studied the thermal dissociation of heavy oils. W. Dozier (Exxon) has used SANS to study the structure of microemulsions. J. Gregory (Mobil) and C. Glinka (Reactor Radiation Division) have examined the structure of hydrogen-implanted silicon, a material useful for solar cells. H. Hanley and G. Straty (NBS-Boulder) have studied concentrated latex solutions and supercooled glycerol. C. Glinka and L. Sander (Center for Analytical Chemistry) have continued their study of the structure of bonded phases in porous silica. R. Slade (U. Exeter, England) used SANS as a tool to study the porosity of clay materials.

SANS has continued to prove a vital tool for probing the microscopic structure of amorphous magnetic systems. J. Rhyne (Reactor Radiation Division) and M. Spano (Naval Surface Weapons Center) have studied a wide range of concentrations of various amorphous rare earth-iron systems, examining the competition between exchange and anisotropy which leads to the formation of an intriguing low temperature state, the correlated spin glass. Rhyne and G. Fish (Allied Corporation) have measured the short correlation ranges in amorphous Fe-Zr resulting from the competition between ferromagnetic and antiferromagnetic exchange interactions. In an international collaboration, Rhyne and B. Barbara (CNRS, France) have probed the critical behavior resulting from the presence of random exchange and anisotropy fields in amorphous $Dy_xGd_{1-x}Ni_2$ alloys. In a different area, J. Lynn, W.-H. Li and Q. Li (U. Maryland) have examined the new high

temperature superconductor $ErBa_2Cu_3O_7$ (T_c = 95K) and found evidence for the screening of paramagnetic fluctuations by the superconducting electrons, the first such observation in a superconducting rare earth system.

K. Hardman-Rhyne (Ceramics Division) continued to take advantage of the non-destructive interaction of neutrons with samples to evaluate the structures of calcinated Green State alumina and alumina powder, as well as the porosity of a variety of systems, including slipcast alumina sheets and, with T. Coyle (Ceramics), sol-gel networks. Hardman-Rhyne also used SANS to study the effects of stress in causing microcracks in composites and creep cavitation in siliconized SiC.

SANS has also been of great utility in the characterization of metallurgical samples. B. Mozer (Reactor Radiation Division) and R. Shull (Metallurgy) have compared the microstructures of amorphous metallic alloys prepared by different techniques. E. Case (Michigan State U.) has examined the microstructure of boron-doped Ni_3Al as part of an investigation of embrittlement mechanisms. R. Fields (Fracture and Deformation Division) and R. Reno (U. Maryland) have also used SANS to study the precipitation of Cu-rich particles and austenite in aged A710 steel. Finally, R. Odette, J. Fint and D. Klingensmith (U. California-Santa Barbara) made strenuous efforts to push the background of the SANS instrument to record-low levels in their attempt to observe small structural changes related to embrittlement in irradiated pressure vessel steels.

Several research groups have taken advantage of SANS to study the structure of biological systems. D. Engelman and J. Flanagan (Yale U.) examined the unfolded states of proteins with altered amino acid sequences in order to understand how one-dimensional amino acid chains fold to produce three-dimensional proteins. As part of a continuing program, S. Krueger (U. Maryland) and R. Nossal (National Institutes of Health) have studied neurosecretory vesicle systems in an effort to understand the role of proteins and protein interactions in their function.

Finally, as part of our theoretical effort in this area, a significant advance has been made in the treatment of small angle scattering from bicontinuous morphologies that bear on an understanding of microdispersed materials, such as porous glasses and microemulsions. The new approach develops a rigorous mathematical analysis of a computer simulation technique, thus accurately predicting the scattering from topologically realistic three-dimensional multi-phase interfaces.

COLD NEUTRON PROJECT

The goal of this program is to develop a Cold Neutron Research Facility at the NBS reactor. This facility will consist of a large volume D_2O ice cold neutron source in the reactor, which will be viewed by seven neutron guides and one beam port. The neutron guides, each of 15 X 6 cm^2 cross-section, will transport low energy (cold) neutrons into a new experimental hall of dimensions 200 feet by 100 feet, providing space for 15 new experimental stations. Additional support space of 15,000 ft^2 will also be provided. Funding for this program began in FY 87, and five new staff members have been brought on board. The total construction cost is estimated at $25 M over five years.

FY 87 Significant Accomplishments

- o Testing of the cold neutron cryostat outside of the reactor was completed. The cryostat was installed in the reactor, and the first startup tests have been completed.

- o A detailed design of the new building was completed, and a solicitation of bids was issued. Bid opening is scheduled for October 5, 1987.

- o The preliminary design of the cold neutron guide tube network was completed, and a request for proposals was issued. The proposals have been evaluated, and a contract will be signed early in FY 88.

- o The conceptual design of the first four new experimental stations was initiated, and some detailed design was begun.

- o A study of advanced neutron optical devices was begun, and results have been obtained for various focusing devices (e.g., elliptical and toroidal mirrors, supermirrors). A request for proposals for advanced R&D on special thin film coatings was issued.

- o A project to develop better neutron monochromators was begun in cooperation with the Institut Laue Langevin in Grenoble, France.

1. Cold Neutron Source

R. S. Carter, J. M. Rowe, P. A. Kopetka and R. S. Williams

The use of cold neutrons (i.e. neutrons with wavelengths and energies characteristic of temperatures ≤ 60 K) is important to many areas of materials science, including the study of microstructure in alloys, polymers and polymer blends, and biologically significant molecules; the study of the motions of large molecules and molecular assemblies; the use of depth profiling to study sub-surface impurities and dopants; and the study of the structure and dynamics of novel materials. The normal reactor moderator produces a spectrum in which the cold neutrons are only 1 % of the total number. However, by replacing a portion of the normal moderator with a cryogenically cooled moderator, this fraction can be increased substantially. The NBSR was designed with a large volume suitable for the insertion of a cold neutron source, and we are installing a 36 cm diameter block of D_2O ice cooled to 30 K into this volume. The first of the two cryostats, modified to increase the cooling flow through the moderator was

received in November 1986. Before installation in the reactor, it was extensively tested for leak tightness, structural integrity, and cooling flow rate. Ice was made in it a total of five times. The final time included a complete test with the cryostat connected to the refrigerator. The cryostat, with ice in it, was successfully cooled to 25 K. Since it was not in the reactor, it had no significant heat load, so the heat load was simulated using auxiliary heaters built into the refrigerator. The system was run for several days with a simulated heat load of 1100 W and the cryostat at 25 K. The expected heat load in the ice from gamma-ray heating is about 800 W, so the system appears to have adequate cooling capacity.

The cryostat and its bismuth/lead gamma-ray shield was installed in the reactor during the summer. Once again, ice was made in the cryostat, but D_2O was used for the first time. The ice making proceeded smoothly and the ice was cooled to 25 K and maintained there for several days with the reactor shutdown. This was the last of the "dry runs," and we are now (September 1987) awaiting reactor start up to begin the final cold source startup tests. These will measure the release of radiolytic gas as the ice warms up after various periods of reactor operation. Measurements will also be made of the cold neutron intensity and spectrum during these tests. The first tests were satisfactorily completed on September 14, 1987, at 5 MW reactor power.

2. Instrumentation and Facility Design

J. M. Rowe, T. Udovic, I. G. Schroder, C. J. Glinka, G. Greene, J. Larock, and D. Fravel

When the cold source is operational, it will be viewed by two beam tubes at which a Small Angle Neutron Scattering (SANS) spectrometer and a Time-of-Flight (TOF) spectrometer are installed. At the same time, we are constructing a new research facility of 20,000 ft^2 (200' X 100') adjacent to the north face of the existing reactor confinement building, and an additional 16,000 ft^2 of support space (laboratories, mechanical equipment and offices) adjacent to the north end (A wing) of the existing office/lab building. The detailed design of this work has been completed, and a solicitation package for bids has been issued. Bids are due by October 5, 1987, and construction will begin within 60 days of that date. The new facility will provide space for at least 15 new experimental stations, of which one-third will be built and operated with non-NBS funds. An agreement has been started with Exxon Research and Engineering for development of one station, and active negotiations for development of another four are actively underway.

In order to make use of the cold neutrons from the cold source, a series of seven "neutron guides" will be installed to conduct neutrons from the source to the new experimental hall. These guides consist of thin layers of nickel deposited on optically polished glass, and provide beams of large cross-section (15 X 6 cm^2) in the new hall. These beams can then be used for the fifteen new experimental stations. In order to increase intensities, the guides will be coated with a special nickel isotope, or possibly with the special "supermirror" coatings discussed below. The conceptual design of the guide network is complete, and detailed design and construction will begin in the fall of 1987.

We have begun the conceptual design of the first four stations to be installed in the new facility--one SANS, one TOF, one triple-axis spectrometer, and one back-scattering spectrometer. In addition, an extensive research and development project on advanced neutron optical devices has been initiated. This project is based upon two major areas--first, neutron focusing devices (mirrors, converging guides, etc.) and second, improved neutron guide materials. In the first area, we have developed Monte Carlo computer codes to simulate various geometries, including realistic manufacturing problems (surface roughness, precision of machining). In the second area, we are cooperating with Oak Ridge National Laboratory and Brookhaven National Laboratory on the development of "supermirrors," which are essentially neutron guides with four to nine times the total intensity of the normal nickel guides referred to above. These devices can be used to enhance total intensity, to focus beams, and to select neutrons of a given spin state (polarized beams). We are also developing a new test station at BT-7 to allow testing of devices as they are made. If this project is successful, at least some of the seven neutron guides will be coated in this way, providing the better cold neutron intensities than have been produced thus far worldwide.

INDEPENDENT PROGRAMS

The two major independent (non-collaborative) Bureau programs using the reactor are nuclear methods group in the Center for Analytical Chemistry and standard neutron fields for neutron flux calibration and materials dosimetry in the Center for Radiation Research. These programs will be summarized here. The major non-NBS independent programs were summarized under the irradiation services provided by Reactor Operations.

FY 87 Representative Accomplishments

- As part of the joint NBS/FDA/USDA/IAEA study of trace elements in the human diet, a total of 30 biologically important minor and trace elements have been measured on a total diet material. A second diet material is now in preparation, which will be developed as an NBS Standard Reference Material.

- In many applications, solid state track recorders require fissionable deposits of very low mass, as, for example, in the monitoring of 18-month-long power reactor fuel cycles. A cooperation has begun with Westinghouse Corp. to provide mass assay of deposits of three fissionable isotopes in the picogram and sub-picogram range. The accuracy goal is a few percent! The quantity measured is the total number of fissions which take place in the SSTR deposit during a high fluence thermal or fission spectrum irradiation. The final mass is established relative to FIMS, the NBS set of Fissionable Isotope Mass Standards.

Nuclear Methods

R. Fleming, Leader

The development and application of nuclear analytical techniques for greater accuracy, higher sensitivity and better selectivity are the goals of the Nuclear Methods Group. A high level of competence has been developed in reactor-based activation analysis, which includes instrumental and radiochemical neutron activation analysis (INAA and RNAA), and in LINAC-based activation analysis using photons (PAA). In addition, the group has a unique capability in neutron beam analysis with both prompt gamma activation analysis(PGAA) and neutron depth profiling (NDP). The NDP technique utilizes prompt charged particle emission to determine elemental distributions within the first few micrometers of a surface while the PGAA technique utilizes prompt gamma-ray emission to measure the total amount of an element in a sample, regardless of its distribution. These techniques provide an arsenal of tools to address a wide variety of analytical problems in science and technology.

The activities of the past year have been highlighted by the initiation of the National Facility for Cold Neutron Research to be established at NBS during FY89. The Group's involvement includes the design and construction of second generation instruments for prompt gamma activation analysis and for neutron depth profiling. In addition a facility is envisioned at which we will study the techniques for focusing neutron beams to increase the neutron intensity on a point. The combination of intense focussed beams applied to the existing analytical methods could result in greatly enhanced measurement capability.

The Group's contribution to the certification of Standard Reference Materials is illustrated by the multielement measurements done on the SRM Filter Paper Blanks, Bovine Serum, River Sediment, and Creatinine. In addition, individual elements have been analyzed in the SRM Cholesterol, Monel, and the Depth Profiling Standard. The extension of the fast neutron activation analysis developed for silicon determination is being made to nickel determination, quantifying the cobalt-58 activity produced by the (n,p) reaction on nickel-58.

The Biomonitoring Specimen Bank Research Project continued its support for other agencies' monitoring programs. These included the EPA human liver project, the NOAA National Status and Trends program, the NCI Micronutrient program, the IAEA/NBS/FDA/USDA Total Diet Study, and most recently, the NOAA Alaska Marine Mammal Project. Research has centered on banking protocols and improved analytical methodology; for example, the determination of vanadium at sub-ppm levels in fish livers. Our participation in intercalibration exercises with the project participants and the development of marine QA materials helped enhance the quality of the analytical results used in the assessment of the Nation's environmental health.

Bioanalytical research focused on the determination of metal species in various materials. Elements at trace and ultratrace levels have been determined in separated proteins and other macromolecules. The use of autoradiography to determine phosphorus has been added to the INAA and RNAA techniques. The occurrence of inorganic and organic compounds of tin in marine tissues is also being studied.

The strong interaction with industrial scientists using neutron depth profiling, prompt gamma activation analysis, and neutron activation analysis has continued with a growing number of guest workers, research associates, and joint publications. The longterm NDP study of the mobility of helium in nickel has been completed. The measurement of distributions of lithium and boron in metals, glasses, and polymers continues to produce important results.

The joint NBS/FDA/USDA study of trace elements in human diet, sponsored by the International Atomic Energy Agency, has completed its third year. A total of 40 minor and trace elements have been measured on the the total diet material. The preparation of USDIETS II, III, and IV has been completed, and analysis of these and other diets received from countries participating in this global study is in progress. In addition, a candidate SRM mixed diet reflecting the US dietary matrix is under preparation.

During the coming year the Group will continue to improve the accuracy and sensitivity of nuclear methods as applied to elemental measurements. Problems to be addressed include those inherent in sample preparation, irradiation, radiochemical separation, counting and data reduction, with the goal of minimizing and quantifying the various sources of random and systematic errors in analysis by nuclear methods.

Ronald F. Fleming,Group Leader; Thelma A. Allen, M.James Blackman, Latane E. Brackett, Donald A. Becker, R.Gregory Downing, Kathleen A. Fitzpatrick, Brent L. Grazman, Robert R. Greenberg, G.Venkatesh Iyengar, John K. Langland, Richard M. Lindstrom, Bruce R. Norman, Craig A. Stone, Susan F. Stone, Theresa M. Sullivan, and Rolf L. Zeisler.

Neutron Field Standards and Dosimetry

J. Grundl, Leader

The Neutron Dosimetry Group of the Center for Radiation Research (CRR) is engaged in the development and the application of well-characterized neutron fields as permanent irradiation facilities for neutron dosimetry standardization, for neutron detector development and calibration, and for reaction cross section measurements. Strong interactions with outside organizations, both in the federal and private sections, are important programmatic elements. The condensed list of technical activities given below includes only those projects which involve facilities at the reactor.

Neutron Personnel Dosimetry

In neutron personnel dosimetry, the Neutron Dosimetry Group's activities include the use of the NBS standard neutron fields for routine calibrations of health physics instrumentation, for development and testing of new types of instrumentation, and for quality control of production instruments. In the area of dosimetry, methods research, a tissue equivalent proportional counter system (TEPC) is under development to determine neutron dose as a function of energy deposited.

1. Purity of the filtered beams. In response to questions concerning the purity of the NBS 2 keV filtered beam, the neutron spectrum was carefully remeasured using a He-3 spectrometer with a new low-noise preamplifier and pile-up rejection circuitry. Newly observed lines between 70 keV and 550 keV amount to only about 2% of the primary 2 keV line. The pulse height spectrum shows that by far the cleanest 2 keV beam at any reactor facility in the world is at NBS. The 144 keV beam also has been improved recently by increasing the amount of Ti in the filter.

2. Quality assurance and type testing. The filtered neutron beams, together with the beam at the thermal column, continue to be used for testing radiation protection instrumentation. Included this year were quality control measurements on neutron area monitors for the Naval Surface Weapons Center, dosimeter irradiations as part of the Department of Energy Laboratory Accreditation Program (DOELAP), and testing of a new type of neutron detector developed at the Chalk River Laboratories. The latter testing, done in collaboration with several other laboratories in the United States and England, forms the basis for a paper to be given at the Sixth Symposium on Neutron Dosimetry to be held in Neuherberg, Germany, in October 1987.

Dosimetry for Materials Performance Assessment

Exposure to neutrons can cause critical performance degradation in materials as different as a silicon wafer and an 8-inch thick plate of steel. Dosimetry methods for assessing the degradation of materials in high-intensity neutron fields are equally diverse. The Neutron Dosimetry Group provides some form of measurement assurance, standardization, methods development, and/or advice for nearly all of the approaches to materials neutron dosimetry pursued in the United States. Because of assorted

commercial interests, foreign involvement is also a strong component of this activity.

1. Neptunium neutron fluence standard. For the first time an Np fluence standard was prepared with sufficient activation to allow the 30-year ^{137}Cs activity generated by the Np fission reaction to be calibrated for dosimetry. The Np fission detector is a uniquely effective threshold detector for establishing a neutron exposure component below 1 MeV, and the ^{137}Cs fission product activity is especially useful for monitoring long-term exposures.

2. Pulsed reactor dosimetry. The Nuclear Effects Laboratory at the White Sands Missile Range has requested help in developing an in-house active fission rate measurement capability connected to NBS standard neutron fields. Plans have been formulated for initial on-site measurements in early FY 88. Fission rate measurements at this laboratory are used to establish neutron dose for electronic components exposed to leakage neutrons from the White Sands fast burst reactor.

Benchmark Neutron Field Facilities and Fission Rate Measurement Capability

The foundation of the Neutron Dosimetry Group's service to nuclear technology are the well-specified neutron fields that are maintained as permanent irradiation facilities mainly at the reactor. Ten separate irradiation sites at NBS provide certified fluences of thermal neutrons (beam or isotropic), of keV neutrons (filtered beams), of sub-MeV distributions (ISNF and the D_2O Moderated Cf Fission Source), or of pure fission spectrum neutrons (Cf or U-235 with fluence rates of up to 4×10^{10} n/cm^{2}s). Complementing the neutron fields, but with independent applications of its own, is the NBS fission rate measurement capability. At the center of this capability are the set of fissionable isotope mass standard (FIMS) and the double fission chambers in which they are used. Passive and active neutron detectors of all kinds are exposed to these neutron fields for calibration, for measurement assurance projects, for cross section measurements, and for development of new measurement techniques.

1. Facility Improvements

- An in-depth review and reworking of the calibration base for the U-235 cavity fission source has begun. The complicated path by which the absolute fission neutron fluence rate is established will be set out more explicitly and calculations of irradiation fluences will be handled with computerized spread sheets.
- A new method for monitoring ISNF irradiation fluences has been perfected. It involves neutron fluence transfer from the Cf fission source via fission product activation counting of U-235.
- The calibration documents SP250-13 covering activation foil irradiations at the Cf fission neutron Irradiation Facility and SP250-14 for activation foil irradiations at the U-235 Cavity Fission Source were completed and published.

2. Fission Chamber Monitors.

Enlarged versions of the NBS double fission chamber were designed and constructed for use as the primary monitor for exposure rooms at the AFRRI TRIGA reactor in Bethesda. This new monitoring scheme, which replaces existing ion chambers, will provide an unambiguous response to neutrons in the MeV range for biological specimen irradiations at high fluence rates.

Integral Neutron Cross Section Measurements

The standard and reference neutron fields, together with the fission rate measurement capability and the nearby facilities of the Radioactivity Group, conspire to make NBS an excellent place to measure absolute reaction-rate cross sections. As a base program this activity has been pursued for a long time, generating influential data in reactor physics as well as neutron dosimetry.

1. Nb(n,n^1) for High-Fluence Materials Neutron Dosimetry.

The NBS cavity fission source at the reactor was the site for a DOE sponsored, multilaboratory cross section measurement that included substantial international participation. Nb is an up and coming dosimetry detector with a 16 year half-life that is attractive for long-term sub-MeV neutron exposure monitoring, but not so attractive for differential cross section measurement. Hence, for this detector, a fission-spectrum-averaged cross section is of particular importance. The results of this work were reported at the Sixth ASTM/EURATOM Reactor Dosimetry Symposium in June, 1987.

2. Photofission for Correcting Fission Detector Responses.

The problem of photofission interference in fission detectors has been around for a long time. To check existing corrections, photofission cross sections in four fissionable isotopes were measured in the new neutron driven gamma ray source designed and constructed by the Neutron Dosimetry group in cooperation with the SCK/CEN laboratory in Belgium. For these measurements, the source was operated first with a cadmium and then with an iron converter. The data obtained will be used to validate and/or adjust calculated correction methods, notably, those developed by C. Eisenhauer at NBS. The measurements were carried out in a cooperation headed by Prof. T. Williamson of the University of Virginia and were reported by him at the ASTM/EURATOM Reactor Dosimetry Symposium in June.

3. Capture Reactions in ISNF.

Two carefully planned irradiations of 20 activation detectors were carried out in the Intermediate-Energy Neutron Field (ISNF). The purpose was to extend previous capture cross section measurements to include Na, Ag, and Sc, all important reactions for reactor physics applications. Major measurement issues were the perfection of a new method of neutron fluence transfer and corrections for resonance self-absorption. Executing and documenting the irradiations was largely done under contract by a retired group member.

Special Projects

The unique irradiation facilities and measurement capabilities in the Neutron Dosimetry Group coupled with the wide range of institutional involvements characteristic of neutron dosimetry in practice continues to bring on small worthwhile projects and unavoidable responsibilities.

1. Neutron Lifetime Experiment.

   In the measurement proposed by the NBS Quantum Metrology Group, the Neutron Dosimetry Group will determine the average linear density of neutron beam downstream from an electromagnetic proton trap. Preliminary experiments carried out this year explored the possibility of using a $^{10}B(n,\alpha,\gamma)$ coincidence technique for absolute thermal neutron counting. More than promising results were obtained and reported in a paper to be published in the Journal of Radioanalytical Chemistry. A visiting scientist from the Univ. of Sussex in England participated in this work.

2. LiF Chips for Megagray Gamma Dosimetry.

   The new LiF Chip dosimeter (see Tech. Act.-86) has been tested for neutron sensitivity this year by exposure in the U-235 Cavity Fission Source. In the course of this work, a new color center was explored and the sensitivity of the dosimeter was extended to the 10 MGy range. In a typical application, the correction for neutron response will be 15 to 20%. Special LiF-glass encapsulation for the dosimeters are being fabricated prior to shipment to Babcock & Wilcock, Co., who have supported this R&D effort. The calibration work has been done in cooperation with the X-ray Physics Group and the results were presented at the ASTM/EURATOM Reactor Dosimetry Symposium in June, 1987.

3. Sixth ASTM/EURATOM Reactor Dosimetry Symposium on Reactor Dosimetry.

   The sixth symposium in this biennual series was held this last June in the U.S. and two group members were strongly involved in preparing and running what has become a rather large international gathering.

OUTPUTS/INTERACTIONS

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Industrial and Academic Interactions

As a national center for the development and application of neutron methods in condensed matter and materials science, chemical analysis and radiation standards, the Reactor Radiation Division currently has direct interactions and cooperative programs with 37 universities, 23 industrial and 18 foreign laboratories. A few examples of these many interactions are:

- An agreement is in place between NBS and Exxon Research and Development Corporation to jointly develop and operate a world-class small angle neutron scattering spectrometer at the NBS cold neutron source. Active work is underway in the design and construction of this facility. Cooperative research efforts on the existing SANS instrument at the reactor include work on wetting in microporous media and micellar systems.

- An intense cooperative research effort was organized early in 1987 with several groups at AT&T Bell Laboratories on the systematic studies of the structure and properties of the new class of high-temperature superconductors.

- The Reactor Radiation Division's Crystal Data Center is engaged in a number of interactive links including joint development and distribution to U.S. science and industry of evaluated crystal data with the International Center for Diffraction Data. The Data Center also has long-term agreements in place with crystal data programs in Canada, Great Britain, and Germany to jointly develop and share critical data on the structure of materials.

- The Neutron Scattering Group has in place wide collaborative research with the University of California (Santa Barbara) involving neutron inelastic scattering, neutron diffraction, and SANS studies of catalysts, non-linear optical materials, and radiation damage.

- Extensive cooperative research efforts with the Physics Department and Materials Research Laboratory at the University of Illinois and with Michigan State have continued during the past year to include the first neutron diffraction work worldwide on a new class of layered magnetic materials and research on new kinds of metal-molecular complexes created within the layers of oriented graphite and in pillared clays.

- Cooperative Research Program with the Department of Physics and Astronomy of the University of Maryland. Under the program RRD staff are engaged with Maryland scientists in joint research on magnetic materials, superconductors, catalysts, biological materials, and in the development of state-of-the-art polarized neutron scattering instrumentation. Some of this research is carried out jointly with scientists from industrial labs.

- Collaborative research activities continue with GTE Research Laboratories on the application of neutron diffraction and small angle scattering techniques to study the structure, residual stress, and microstructure in new ceramics used for electronic and auto-engine components.

Associated Activities

During the past year, scientists in the Reactor Radiation Division delivered some twenty-five invited lectures in the U.S. and abroad. The Division is also hosting the national meeting of the U.S. Test, Research, and Training Reactors this year. Neutron Scattering Group scientists have also received, along with French and German colleagues, several NATO grants to stimulate cooperative research in such areas as magnetism and hydrogen in metals.

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This report summarizes all those programs which depend on the NBS reactor. It covers the period from October 1, 1986 through September 30, 1987. The programs range from the use of neutron beams to study the structure and dynamics of materials through nuclear physics and neutron standards to sample irradiation for activation analysis, isotope production, radiation effects studies, neutron radiography and nondestructive evaluation.

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